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DIFFERENTIAL SCANNING CALORIMETER DATA ACQUISITION SYSTEM

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The kinetics of polymerization reactions are studied in our laboratory using differential scanning calorimeter (DSC) data at various temperature scanning rates and analyzed by a computer code based on a theory developed and expanded in this laboratory. This created the need for an efficient data acquisition system with a variable and accurate programmable data collection rate. This report discusses one such successful data acquisition system using a Bascom Turner recorder (BTR). It is a combination chart recorder, calculator, floppy disk storage device, and interface allowing data to be transferred to a printer, CRT, or computer. Step-by-step procedures are presented.

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FOREWORD

This report was prepared by the University of Dayton Research Institute, Nonmetallic Materials Department, Polymer Group. This work was initiated under U.S. Air Force Contract No. F33615-81-C-5019, "Polymeric Materials for Advanced Aircraft and Aerospace Vehicles", with Dr. Donald R. Wiff as the principal investigator. It was administered under the direction of the Materials Laboratory, Air Force Wright Aeronautical Laboratories, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio with Dr. T. E. Helminiak as the contract monitor. The author of the report was Ms. Marlene Houtz, University of Dayton Research Institute.

This interim report covers research conducted from August 1982 to June 1983.

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

SECTION I
INTRODUCTION

The kinetics of polymerization reactions are studied in the Air Force Wright Aeronautical Laboratories (AFWAL) Polymer Laboratory, using data obtained through differential scanning calorimetry (DSC) and subsequently analyzed by application of a computer code based on a theory developed[1] and expanded in this laboratory. This creates the need for an efficient data acquisition system with a variable and accurate programmable data collection rate. The data format must be in a form that can be transferred easily to a computer disk file for permanent storage and easily retrieved when needed for computer processing.

Until recently, data was obtained by a multicomponent system which consisted of a Hewlett Packard 3480D digital voltmeter (DVM), a Hewlett Packard 2570A coupler controller (CC), and a teletype unit with paper tape output capabilities. This system acquires an analog signal from the DSC (Perkin Elmer DSC-II), converts that signal to a digital form through the DVM, carries the signal through the coupler controller at a programmed data rate to a teletype unit which prints the data while simultaneously punching the information on paper tape. Additionally, an analog signal from the DSC sends the information to a strip chart recorder where the exothermic and endothermic transitions can be visually monitored. The multicomponent system diagram is shown in Figure 1.

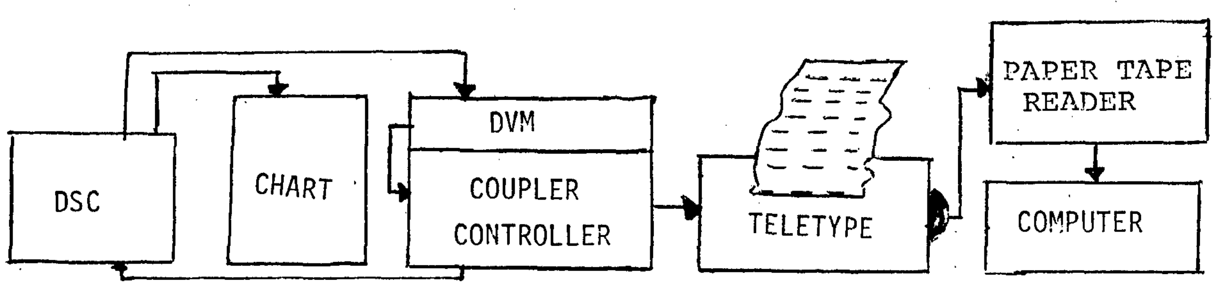

Figure 1. Coupler Controller Data Acquisition.

The CC initiates the DSC to heat at a preset programmed rate from an initial temperature. The DVM picks up the changing mV signals while the CC sends these signals to the teletype at a continuous constant data rate. The range limits of the CC are 0.6 to 99.9 seconds/data point which is practical for this application.

The CC method is efficient and accurate. However, there are some drawbacks which resulted in its replacement by a Bascom Turner 4120 recorder (BTR).

(1) The CC method has the disadvantage of being multi-component with multisource serviceability. Service on one component was available rapidly, while another component could take one month or longer. The circuit boards of the CC are no longer available for replacement, so repair, if ever necessary, could not be accomplished.

(2) The data collected on paper tape has to be read through a paper tape reader located outside of the laboratory facility. The data was stored in one computer system and then transferred to another computer system for final permanent file storage. Additionally, paper tape data storage is cumbersome and outdated as floppy disks and magnetic tape cassettes are in current use with computer systems.

The following sections will discuss some of the features of the BTR, its operation, calculations, computer access, and data transfer. A comparison of the two systems will follow in Section VI.

SECTION II
BASCOM TURNER RECORDER FEATURES

The Bascom Turner recorder (BTR) is a combination chart recorder, calculator, floppy disk storage, and has interface options that allow data to be transferred to a printer, CRT, or computer. It is easy to set-up, operate, maintain,and occupies little space on a benchtop. The recorder has two input channels with a range of 10 mV to 10V. Both channels have a data collection rate of .001 to 999 seconds/data point. The analog data is digitized, displayed, and plotted simultaneously. The data collected can be Y-time or X-Y format. A total of 500 data points can be stored on a record and one disk will store 270 records of data. The data format is ±XX.XXX for the range of 10 to 50 mV. Stored data can be recalled and compared with other data, expanded, condensed, integrated, and various mathematical manipulations.

Connecting the BTR to the DSC is relatively simple. The two ordinate (calorimetric) analog signals from the DSC are connected to the top of the BTR into channels 1 and 2 and the appropriate mV signal for each channel is set. It is advised to read the Bascom Turner Manual [2] section on initial operations for complete precautions.

Data acquisition via the BTR is fairly simple. The data collected from the DSC is an analog signal which is stored in a plot buffer, or temporary storage location, and plotted as Y versus time. The analog signal is acquired, converted to digital form, displayed on the recorder and stored in a plot or disk buffer. A schematic diagram of the process is shown in Figure 2. The details on buffer to disk transfer are discussed later in this report.

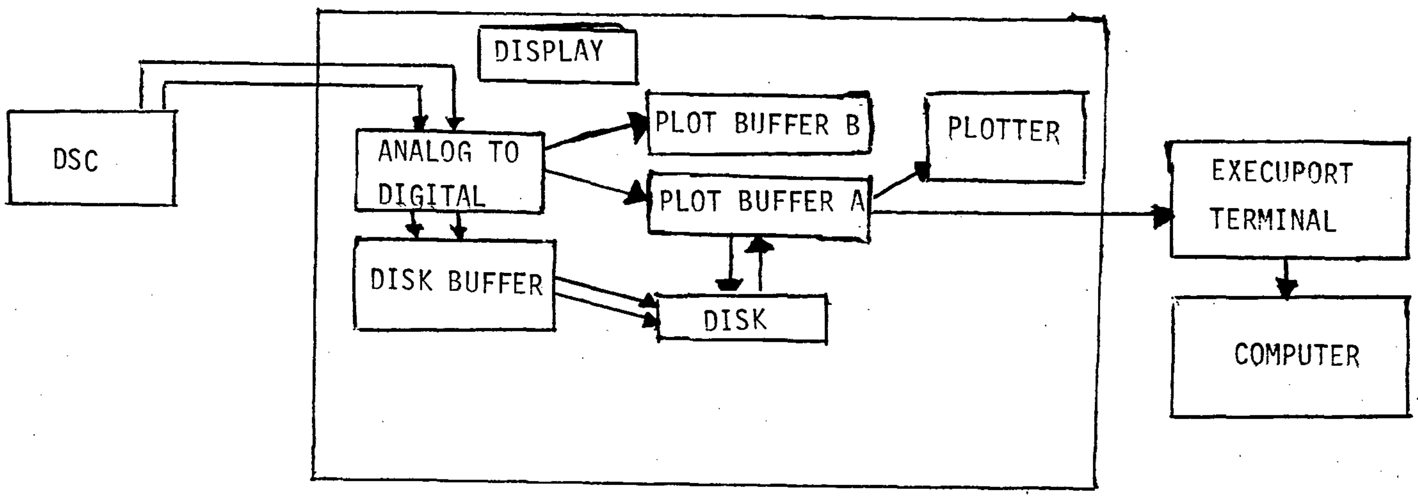

Figure 2. Bascom Turner Data Acquisition System.

SECTION III

OPERATION OF THE BASCOM TURNER RECORDER

A. Initial Operations

1. Load paper by lifting the carriage arm and placing the Z-fold stack on the right hand side.
2. Load program into disk drive until it clicks and close cover.
3. Insert felt tip pen into holder.
4. Power instrument up.

*Note: To protect amplifier, depress zero (green control switch) when a channel is not in use!

Upon power up, the program loads automatically into a memory buffer. Four dots will appear on the display indicating all functions are properly loading. If an error is detected while loading, an error message will appear on the display and the interpretation can be found in the instrument manual. When the recorder is fully loaded and ready, the recorder responds with "Hello" printed on the display.

At this time, data collection via the BTR can begin. Some preliminary commands will align the chart paper and label the incoming data.

B. Sample Data Collection

1. Plot 1 GO — pen places a dot at X=X=0

If paper is not aligned properly use the following commands:

2.	Plot 8	0	move pen .005" in the X direction
	Plot 8	1	move pen .005" in the Y direction
	Plot 8	2	move pen .005" in the -X direction
	Plot 8	3	move pen .005" in the -Y direction exit command with GO

To label the data for ease of identifying

3.	Label 1 XXXXXX GO	This is the date of the experiment
4.	Status 11 n GO Status 12 n GO	Set data rate channel 1 Set data rate channel 2

where $n = 120 \frac{Y}{X} \frac{(\text{final temp} - \text{initial temp})}{(\text{heating rate})}$

n was derived from this equation:

$$n = \frac{\text{msec}}{\text{data point}} = \frac{60 \text{ sec}}{\text{min}} \left(\frac{1000\text{msec}}{\text{sec}}\right) \left(\frac{\text{min}}{X\ {}^\circ\text{C(heat rate)}}\right) \left(\frac{Y^\circ\text{C range scanned}}{500 \text{ data points collected}}\right)$$

Normally for a scan from 323K (50°C) to 723K (450°C) n would be:

Heating Rate (°/min)	n
80	600
40	1200
20	2400
10	4800
5	9600

5. Set channel 1 on 10 mV; channel 2 on 20 mV. This is to ensure against data loss due to off scale or over shoot.

6. ACQUIRE 1 GO (Pen Up) (Do not push heat on the DSC at this time — This is to find where the pen is with respect to desired location. Reposition pen by moving zero control on DSC depending on transition expected.

7. ACQUIRE 9 GO — Stop this acquisition

NOW ACTUAL DATA COLLECTION BEGINS:

8. There are 3 options:

   1. ACQ 1 GO* (pen down) ch 1 collection
   2. ACQ 2 GO* (pen down) ch 2 collection
   3. ACQ 3 GO* (pen up) both channels

9. Note: push HEAT on DSC and GO* on BTR simultaneously.

At this point, data acquisition is in process. If option 1 is used, the data from the DSC will go into Plot Buffer A (PBA) and then plot simultaneously. If the data collection rate is very fast, the plotter will lag behind actual collection; however, it is plotting from the buffer memory so no data is lost. If option 2 is used, the data will go into Plot Buffer A (PBA) and plot. If option 3 is used ch 1 data will be stored in (PBA)

and ch 2 data in (PBB) and the plotter will attempt to plot X-Y which in this case is nonsense as the data desired is Y-t It is for this reason the pen is left up. The desired plot(s) can be obtained in the following manner.

10.	Plot GO	Plot ch 1
	Disk 2 GO	Send this data to disk for permanent storage if desired. A disk address will appear on the display which should be recorded in a notebook.
11.	Cal 22	Exchange ch 1 (PBA) and ch 2 (PBB) data. This is necessary as only PBA will send to disk or plotter.
	Plot GO	Plot ch 2 which is stored in PBA now after the exchange
	DISK 2 GO	Send ch 2 data to disk for permanent storage if desired and record disk address.

SECTION IV

BASCOM TURNER CALCULATIONS FOR KINETICS

One of the calculations necessary to obtain kinetics of polymerization reactions is the subtraction of one data set from another or what is referred to as "baseline subtraction". This subtraction corrects for errors in the baseline slope due to instrument factors and/or sample factors. This subtraction can be accomplished by the kinetics program, however the resultant curve can be seen immediately on the BTR without going through computer processing. Another advantage is the baseline subtracted curve can be scaled up if necessary or compared with another curve previously stored on the disk.

Baseline correction can be done from data stored on disk or with data already in the plot buffer memories. Assuming the original curve 1 is at disk address 111 and the baseline curve 2 at disk address 222, the resultant subtraction is curve 3 as shown in Figure 3. The Bascom Turner commands to accomplish this are:

1.	DISK 1 222 GO	Baseline is copied from disk to PBA if not already there.
2.	CAL 21	Copy baseline to PBB (actually exchange PBA with PBB)
3.	DISK 1 111 GO	Original curve 1 now in PBA (retrieved from disk address 111)
4.	CAL 24 *	Subtract curve 2 from curve 1 or PBA-PBB
5.	PLOT GO	Plot new curve 3 which is in PBA
6.	DISK 2 GO	Store new curve, record address

*Note: this subtraction of curve 1 - curve 2 or PBA-PBB results in the new curve held in memory at PBA. If curve 1 is new data not stored on disk it will be lost as this command overwrites the contents of PBA.

It may be necessary after baseline subtraction to shift the curve's position. In some cases the new curve will appear off scale; however, no data is actually lost as these new values are in the plot buffer memory but exceed the limits of the plotter as set.

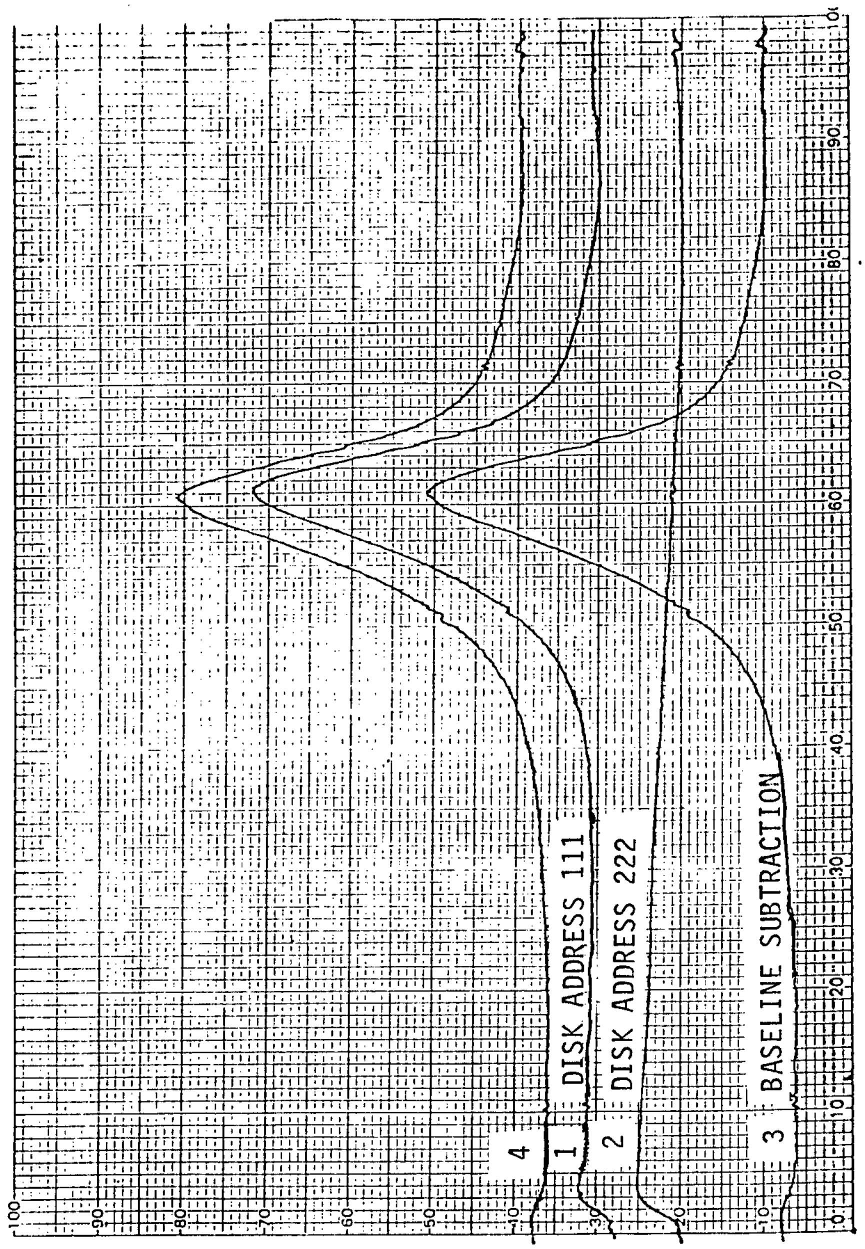

Figure 3. Baseline Subtraction Example.

To shift the curve use the following commands:

7.	CAL 18 XX.XX GO	Add XX.XX to all values of the curve. Shifts Y axis up.
	CAL 18 - XX.XX GO	Subtract XX.XX. Shifts Y axis down.

Curve 4 of Figure 3 is curve 3 plus 3.00 shift upward.

There may be cases where a sample yields a small peak curve and scale-up of this curve will aid the kinetics program in locating the peak and calculating the area under the curve.

8.	CAL 17 XX.XX GO	Multiply curve by XX.XX.

Curve 1 of Figure 4 was multiplied by 3.00 which resulted in curve 2. The curve appears to have gone off scale; however, shifting the curve downward by 3.00 (CAL 18 - 3.00 GO) resulted in curve 3. Notice that scaling up the curve also scales up any noise, baseline slope, and other instrumental factors. Also note that when a curve is multiplied or divided (CAL 17 - XX.XX GO) the appropriate scale factor will need to be corrected for the weight of the sample before running through the kinetics program. In other words, if the sample weight is Y.YY and the resultant curve was multiplied by X.XX, the sample weight will need to be corrected by this factor X.XX(Y.YY). A shift upward or downward of the curve will not affect kinetic calculations provided the curve does not go off scale.

Another feature of the BTR is the ability to integrate a curve. The integral can then be used to find the heat of reaction of a sample. Before actual integration begins, be sure the data has been stored permanently on disk. The commands for integration are:

DISK 1 AAA GO	Retrieve address AAA
PLOT GO	Plot curve

The section of the curve to be integrated must be lowered to zero on the Y-axis. It is not important if other sections of the curve go offscale as only the area to be integrated must be onscale and the beginning and end of the curve must be on zero.

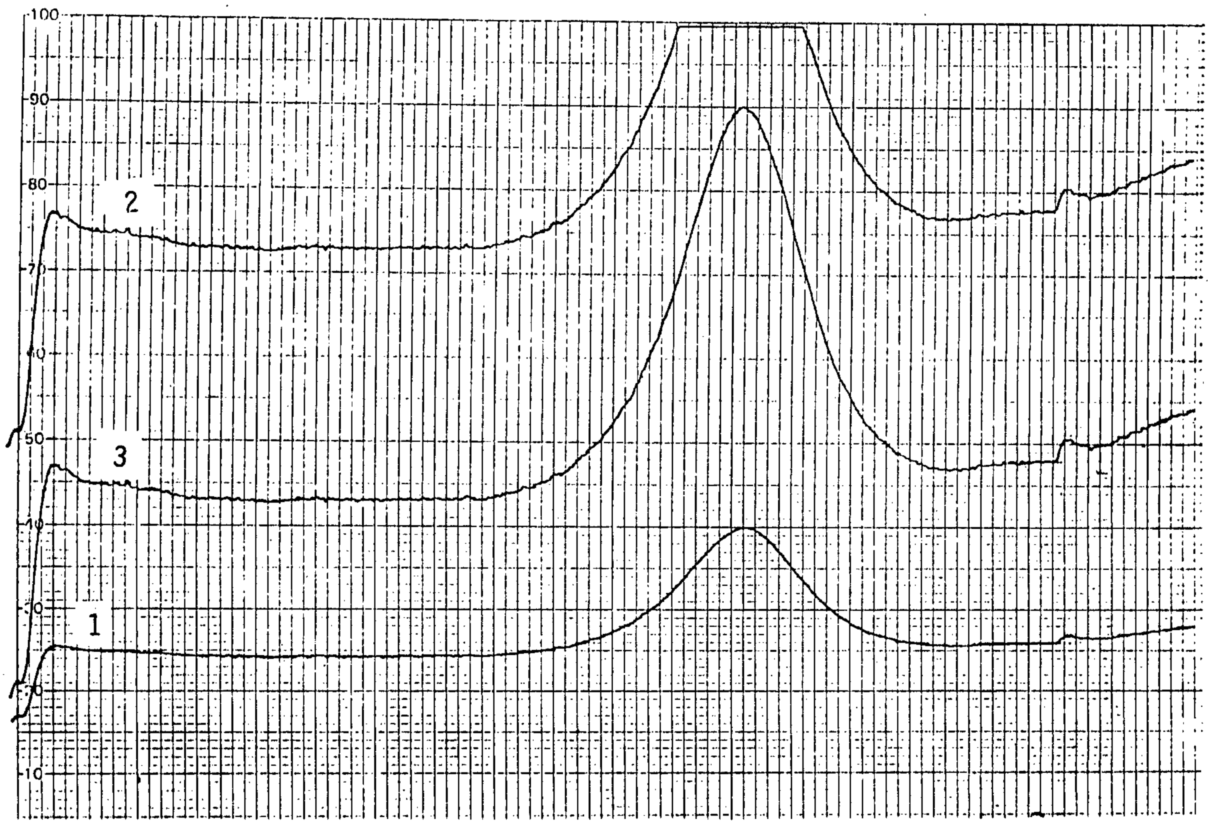

Figure 4. Example of Curve Scale-up.

Curve 1 of Figure 5 is the original curve stored at address AAA. Curve 2 was accomplished by subtracting 1.93 from curve 1 thus lowering the beginning and end of the curve to zero. The command used was:

CAL 18 - 1.93 GO	shift axis by 1.93 downward
PLOT GO	plot new curve
CAL 13 3.5 - 8.00 GO	Integrate between X-axis valves 3.5 and 8.0
PLOT GO	Plot the integral; note the original curve and the new curve will be lost if not stored on disk as the integral is over written in PBA.

The height of the integral $\int ydt$ is then used to find ΔH.

$$\Delta H \text{ cal/gram} = \frac{1}{200} \left(\int ydt\right)\left(\text{—} \frac{\text{mcal}}{\text{sec}}\right)(\text{— data rate})/\text{—mg})$$

For the example in Figure 5, mcal/sec = 20, data rate = 600, weight = 4.15 mg.

$$\therefore \Delta H = \frac{1}{200} (8.5)(20)(600)/4.15 = 121.44 \text{ cal/gram}$$

In some cases, the curve to be integrated will have the end point higher or lower than the beginning. This will cause a problem when integrating as the true area will not be found unless corrections are made. In Figure 6 the beginning of the curve is at 1.3 on the y axis and the end at 0.62. To correct this a generated curve, BASELINE was manipulated to the desired slope by CAL 18 and CAL 17 commands and subtracted from curve 1. The resultant curve 2 has the beginning and end at 1.7 and can be moved to zero (CAL 18 - 1.70 GO) and subsequently integrated as usual.

A quick summary of the commands for the Bascom Turner needed for DSC data acquisition are given in Table 1 and the complete detailed description of all keyboard commands can be found in section 5 of the instrument manual.[2] The aforementioned commands are the basic commands for obtaining data for kinetics.

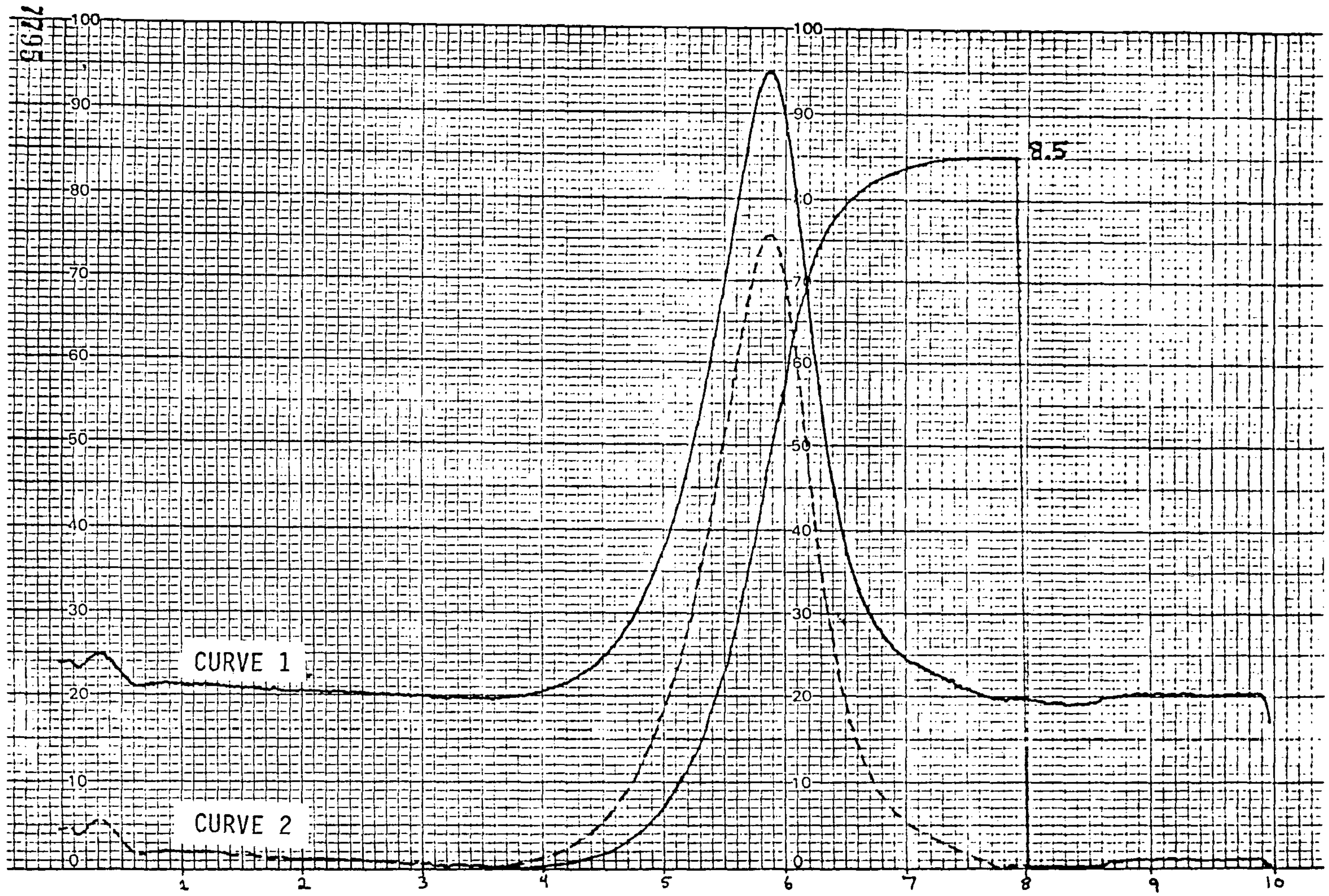

Figure 5. Example of Integration Under a Curve.

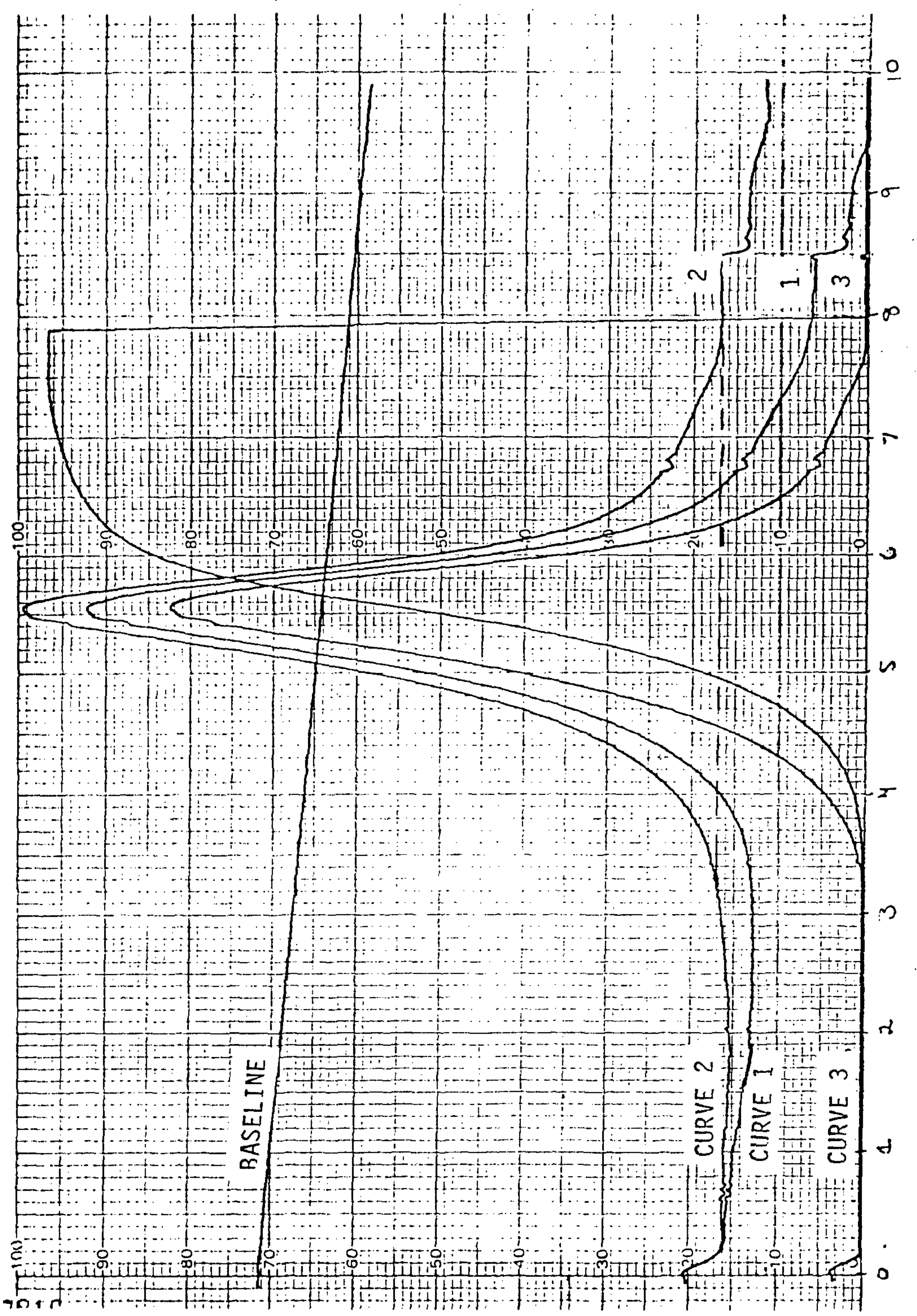

Figure 6. Corrections for Integrating Curve.

TABLE 1

BASCOM TURNER RECORDER FUNCTIONS
NEEDED FOR DSC DATA ACQUISITION

1) PLOT FUNCTIONS

PLOT 1 GO	pen places a dot at 0,0 of the paper
PLOT 8 0 GO	moves pen .005" in the X direction
PLOT 8 1 GO	" " Y "
PLOT 8 2 GO	" " -X "
PLOT 8 3 GO	" " -Y "
PLOT GO	PLOT PBA in Y-t mode
PLOT 5 n GO	move paper forward n pages or backward -n pages.

2) ACQUIRE FUNCTIONS

ACQ 1 GO	collect data channel 1 store in PBA and plot in Y-t
ACQ 2 GO	collect data channel 2 store in PBA and plot in Y-t
ACQ 3 GO	collect data from both channels store ch 1 in PBA and ch 2 in PBB
ACQ 9 GO	stop acquisition and plotting

3) DISK FUNCTIONS

DISK 1 AAA GO	copy from DISK address AAA and store in PBA
DISK 2 GO	store PBA on disk at address displayed
DISK 3 AAA GO	ERASE ADDRESS AAA; data in PBA will be lost.
DISK 5	Display available disk space

4) CALCULATIONS

CAL 13 X.XX - X.XX GO	INTEGRATE ($\int ydt$)
CAL 15	smooth curve
CAL 17 XX.XX GO	multiply (scale up) by XX.XX
CAL 17 -XX.XX GO	divide by XX.XX

TABLE 1 (continued)

BASCOM TURNER RECORDER FUNCTIONS
NEEDED FOR DSC DATA ACQUISITION

CAL 18 XX.XX GO	add XX.XX to curve, shifts Y axis
CAL 18 -XX.XX GO	subtract XX.XX from curve, shift Y axis
CAL 21	copy PBA to PBB or ch 1 to ch 2
CAL 22	exchange PBA and PBB
CAL 23	add PBA to PBB; result stored in PBA
CAL 24	subtract PBB from PBA, result stored in PBA
CAL 25	multiply PBA by PBB, result stored in PBB
CAL 26	divide PBA by PBB, result stored in PBB
CAL 27	average (PBA + PBB/2), result stored in PBB
CAL 41 GO	send data in PBA to the execuport
CAL 474 GO	set baud rate at 300 for transfer to execuport

SECTION V

SENDING THE ACQUIRED B.T. DATA TO A CDC COMPUTER

The kinetics computer program is currently written in fortran IV and run on a CYBER 750 computer. The bascom turner is connected by cable to an execuport 300 terminal with an acoustic coupler for telephone interface. Some modification was required in the kinetics programs to accept BT format.

Sending the acquired data to a permanent file is relatively simple. First, make sure the cable from the BTR is connected to the execuport at the plug-in marked external device. The cable must be in securely at both sites. Inside the execuport the control parameters are to be set on the following: Mode = line, Duplex = half, char/sec = 30, parity = even, QSL = lower. Now the computer number can be dialed up and at the sound of a high pitch tone the phone can be placed in the acoustic coupler of the execuport. The execuport will light up READY in green.

The following commands are performed through INTERCOM, a system which provides time sharing access to the CYBER computer. The initial steps necessary for access are as follows: The underlined commands are to be keyed into the terminal, those without are the systems response. After each command has been keyed in push RETURN to send the command to the computer. If a mistake has been made push the BACK SPACE key and retype command.

1. At the Terminal

<u>LOGIN, XXXXXXX, ZZZZ</u>	XXXXXXX=problem# ZZZZ = code word system responds with COMMAND mode
COMMAND - <u>EDITOR</u>	Sets up editor mode, systems response..
..<u>F, C=80</u>	Sets format to receive 80 characters
..<u>C</u>	Create a file
100=	Terminal sends out the first number and waits on the BT to send

2. On the BT (assuming disk is in and memory loaded)

CAL 474 GO	sets baud rate at 300
DISK 1 AAA GO	send data set AAA to PBA
CAL 41 GO	send contents of PBA to the CDC

3. At the Terminal

The terminal will start receiving data. The data will appear similar to the following: (A condensed data table appears in Fig. 7.)

```
    ADR     REC     DATE     CHN      TIME      SC     MIN    #PTS   CUR NX
    107     000     112581    1      000600     00     000     500   107

02.825   02.975   03.035    X    X    X    X    X    X    X
03.647      X        X      X    X    X    X    X    X    X
```

After completion of this data set, two dots will appear and the next data set can be sent.

4. On the BT

DISK 1 AAA GO	send another data set to PBA
CAL 41 GO	send this set to CDC

5. At the Terminal

The terminal will start receiving data

A maximum of 3 data sets should be sent at one time as the computer may log out automatically not realizing it is in use. After the 3rd data set:

6. At the Terminal

..=	Exit from create mode. At this point, terminal starts spewing out data with line numbers as the terminal could not keep up with the BT although all data was received. As this data spewing is time consuming, boring, of no value, and wastes paper, it is best to exit from this mode by the following: push excape, then % A. This may need to be repeated until the editor mode (..) is shown.
..REQUEST, A, *PF	Request, any file name A-Z for permanent file space.
..S,A,N,O,A	Save, file A, nonsequential, overwrite, all

03.790	03.775	03.765	03.757	03.772	03.657	03.595	03.527	03.485	03.452
03.460	03.437	03.432	03.430	03.412	03.442	03.422	03.427	03.427	03.412
03.422	03.410	03.412	03.405	03.410	03.417	03.430	03.427	03.417	03.410
03.422	03.420	03.410	03.417	03.420	03.415	03.427	03.427	03.440	03.417
03.417	03.427	03.410	03.427	03.412	03.417	03.412	03.420	03.417	03.410
03.425	03.417	03.417	03.400	03.420	03.407	03.415	03.415	03.415	03.415
03.397	03.405	03.400	03.397	03.397	03.377	03.387	03.402	03.395	03.395
03.390	03.397	03.397	03.372	03.387	03.395	03.397	03.400	03.390	03.377
03.387	03.395	03.387	03.380	03.392	03.365	03.372	03.370	03.372	03.377
03.375	03.380	03.375	03.362	03.365	03.357	03.365	03.362	03.365	03.375
03.352	03.350	03.350	03.372	03.352	03.347	03.327	03.337	03.332	03.310
03.277	03.302	03.267	03.270	03.257	03.222	03.217	03.227	03.202	03.200
03.182	03.172	03.155	03.162	03.157	03.170	03.162	03.167	03.162	03.150
03.152	03.162	03.157	03.167	03.170	03.150	03.170	03.172	03.160	03.170
03.155	03.160	03.160	03.120	03.165	03.157	03.167	03.145	03.155	03.170
03.167	03.155	03.157	03.172	03.172	03.167	03.150	03.145	03.170	03.157
03.177	03.170	03.187	03.175	03.190	03.182	03.180	03.180	03.180	03.182
03.185	03.175	03.180	03.190	03.192	03.197	03.202	03.195	03.200	03.205
03.205	03.192	03.195	03.207	03.212	03.212	03.235	03.237	03.252	03.235
03.245	03.262	03.252	03.277	03.267	03.280	03.272	03.297	03.305	03.312
03.327	03.337	03.350	03.345	03.360	03.352	03.422	03.392	03.392	03.412
03.412	03.432	03.460	03.465	03.460	03.487	03.490	03.502	03.537	03.552
03.557	03.572	03.597	03.610	03.620	03.662	03.682	03.707	03.725	03.750
03.777	03.800	03.830	03.875	03.902	03.927	03.957	03.982	03.990	03.992
04.012	03.965	03.932	03.877	03.857	03.947	04.077	04.250	04.432	04.577
04.695	04.790	04.860	04.947	05.035	05.102	05.180	05.262	05.330	05.400
05.472	05.540	05.600	05.667	05.737	05.797	05.857	05.922	05.972	06.050
06.102	06.115	06.195	06.195	06.307	06.277	06.315	06.310	06.307	06.307
06.282	06.257	06.230	06.187	06.137	06.097	06.025	05.957	05.912	05.825
05.752	05.677	05.597	05.532	05.437	05.375	05.297	05.235	05.147	05.087
05.017	04.945	04.877	04.817	04.755	04.692	04.637	04.580	04.525	04.470
04.420	04.365	04.322	04.260	04.220	04.190	04.155	04.097	04.050	04.012
03.995	03.935	03.910	03.875	03.855	03.820	03.790	03.757	03.710	03.695
03.672	03.630	03.612	03.610	03.567	03.535	03.515	03.500	03.477	03.457
03.425	03.400	03.392	03.365	03.347	03.330	03.297	03.287	03.290	03.262
03.250	03.225	03.207	03.197	03.192	03.177	03.152	03.140	03.120	03.105
03.107	03.080	03.072	03.052	03.050	03.035	03.007	03.012	02.997	02.970
02.982	02.950	02.932	02.947	02.925	02.925	02.920	02.912	02.922	02.877
02.885	02.890	02.862	02.670	02.845	02.847	02.832	02.822	02.832	02.840
02.815	02.800	02.792	02.780	02.767	02.760	02.752	02.750	02.735	02.727
02.745	02.720	02.725	02.710	02.717	02.687	02.677	02.682	02.675	02.652
02.645	02.642	02.640	02.630	02.605	02.607	02.602	02.580	02.592	02.557
02.567	02.562	02.532	02.537	02.520	02.515	02.535	02.515	02.477	02.485
02.465	02.460	02.457	02.442	02.427	02.425	02.402	02.395	02.397	02.382
02.357	02.355	02.340	02.337	02.325	02.305	02.295	02.295	02.277	02.275
02.262	02.252	02.250	02.215	02.207	02.207	02.180	02.165	02.157	02.150
02.127	02.120	02.110	02.112	02.092	02.087	02.055	02.032	02.025	02.015
02.012	02.002	01.977	01.967	01.950	01.935	01.915	01.902	01.885	01.872
01.857	01.850	01.825	01.815	01.787	01.782	01.780	01.755	01.760	01.715
01.730	01.685	01.667	01.665	01.635	01.617	01.605	01.580	01.570	01.565

Figure 7. Bascom Turner Raw Data.

..CATALOG, A, TEMP, CY=1	Catalog file A under the name TEMP at cycle 1
..C	Create the next series of data

7. On the BT

   Repeat steps 4 and 5 until remainder or up to 3 data sets have been sent. Then repeat steps 6 requesting a permanent file with a new name, B, and catalog under CY=2. Repeat for as many times as necessary to send all data needed.

8. On the Terminal

   Assuming all data is sent and stored, it now needs to be merged and the file cleaned up and sample values added.

..E,A,S	Edit file A sequentially
..S,Z,M,N,ALL	Save in Z, merge, nonsequential, all
..E,B,S,	Edit file B
..S,Z,M,N	Save in Z merging nonsequential

repeat until all files are merged in file Z

..E,Z,S,	Edit the new file sequentially
..L,A,/ADR/	List all lines with ADR in them, this will locate the beginning of each data set

```
100=ADR
   =ADR
   =ADR
   =ADR
```

System's respone with numbers to the left of the equal sign representing line numbers where ADR is found

Now list above and below each ADR to locate what is there.

For example:

```
..L, 580,620
580=02.852 03.872 etc.
590=..
600=ADR    REC    DATE   etc.
610=107    000    112581 etc.
620=03.261 04.872 etc.
```

580 will be the end of the previous data set
600 will be the beginning of data set 107 (disk address)
at this point delete lines 590 through 610 as they are not needed for the kinetics program

..D,590,610	Delete 590 to 610
..A,590,2	Add to line 590 in increments of 2 Here is where values for each sample will be inserted
590=	Hit return and this will leave a blank line. This blank line indicates the end of a data set in the kinetics program
592=KINETIC SCAN OF	Title of scan and identifying what sample is
594=XX.XX	Weight of sample in mg
596=XX.XX*	Mcal/sec range on DSC, see note below
598=XX.XX	Heating rate °/min.
600=XXX.X	Starting temperature of DSC in Kelvin
602=X.XXX	Data rate in sec/data pt.
	Note: On BT the data rate has to be msec/data pt. so if the data rate was 600 it will be recorded as 0.6 here.

Repeat listing lines before and after ADR to clean up the file. Insert the appropriate sample weights, heating rates, etc. where necessary. If there appears to be data that repeats zeros at the end of a file, delete those lines with zero. The BTR inserts zeros in a record where no data has been collected.

*For mcal/sec range on 20mV setting multiply the range of the DSC by 2. For 10mV setting the range is that of the DSC setting.

At the end of the file, two blank lines should be inserted. This signals to the program the data set is final and analysis can continue.

..L,L	List last line of file to find end
XXX=..	System response-line #XXX
..L,XX,XXX	List a few lines before to check for zeros in file. If found, delete those lines.
..XXX=	Leaves two blank lines, XXY
XXY=	Must be any number higher than XXX which was the last line of the file.

Now the file is completely edited and can be saved in a permanent file.

..REQUEST,Q,*PF	Request to save the edited version in file Q.
..S,Q,N,O,A	Save this file.
..CATALOG,Q,INPUTDATA, RP=987	Catalog this file, RP=retention period in days

The data sets are now ready to use as input data to the kinetics program.

SECTION VI

COMPARISON OF BT RESULTS WITH THE COUPLER CONTROLLER METHOD

The CC method receives an analog signal from the DSC and converts that signal to a digital form through the DVM. The signal is then picked up by the CC at a programmed data rate and printed on paper and simultaneously punched onto paper tape. A second analog signal is sent to a strip chart recorder.

The BTR receives an analog signal from the DSC which is also converted to digital form. The signal received is stored in a plot buffer memory, PBA or PBB, and is acquired at a programmed data rate. The data stored in memory is then permanently stored on floppy disk. A plot can also be obtained from the plot buffer memory.

The major difference between the two systems is in the CC system. The data is acquired through several components and in the BTR through one component. With the CC system the data must be read through a paper tape reader. The BTR can send the data to the computer through a terminal. The BTR can also manipulate the data mathematically by scale-up, baseline subtraction, or normalize.

The other advantages of the BTR have been stated elsewhere, however, the main advantages is floppy disk storage. This allows 270 data curves to be stored on an eight by eight inch flat magnetic floppy versus punched paper tape which would require considerable more space. Also the ease of data transfer to a computer permanent file is desirable.

A data comparison of the two systems indicate very little difference. The CC method acquires data with values ±XX.XX while the BTR acquires values ±XX.XXX when set on a 10, 20 or 50 mV scale. With both systems a set of calibration data is used to correct for thermal lag, area heat constant, and temperature using data obtained from pure elemental samples such as lead or indium. The Y axis is a mV reading relating to mcal of heat

and the X axis (temperature) are both corrected by the calibration.

A plot of rate of evolved heat in mcal/mg-sec versus temperature in degrees Kelvin is shown in Figure 8 for the two systems. The data sets were not taken simultaneously and thus, the weights of the samples varied. The error in sample weight would make a difference in the mcal/mg-sec Y axis scaling as can be seen. The differences in the X axis (temperature) are due to lag factors from the CC which are now corrected for.

The BTR can be updated to other terminals as the need arises. An execuport terminal was chosen as it was currently available. Also, it can be used with any instrument yielding analog signal output in the range of 10 mV to 10V and up to two channels of input. Additional software options are available as are update program versions. The information given in this report is Version 4.

KINETIC CURVES PLOT

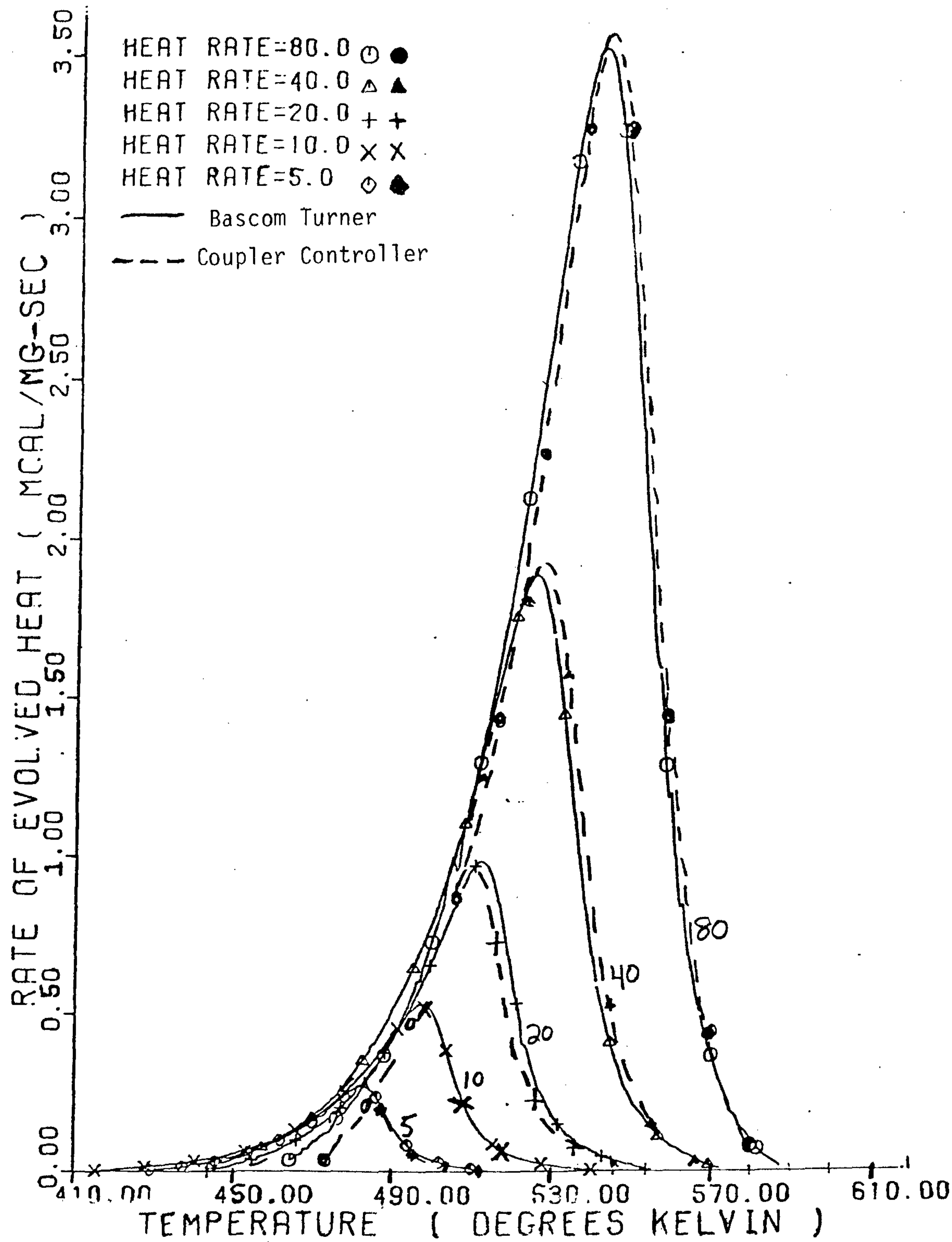

Figure 8. Bascom Turner and Coupler Controller Data.

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